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(54) Plate-cone viscometer with viscosity-dependent regulation of shear rate

(57) For sequential measurement of dynamic viscosity, a plate-cone viscometer with viscosity-dependent regulation of the shear rates has been developed, wherein during measurement the drive motor 4 for the measuring cone 3 is controlled by the output signal from an analog computer 7 included in a regulating circuit, this component linking the electrical signals for the torque u_M and speed u_N continuously and in weighted manner. Moreover, the measuring cone is brought into an optimum position relative to the plate 1 by means of a motor-driven micrometer spindle under microprocessor control for each measurement, the measuring position being signalled by electrical contact.

Using the method of operation according to the invention it is possible to obtain direct comparability with other viscosity measuring systems, e.g. the outflow cup according to DIN 535211.

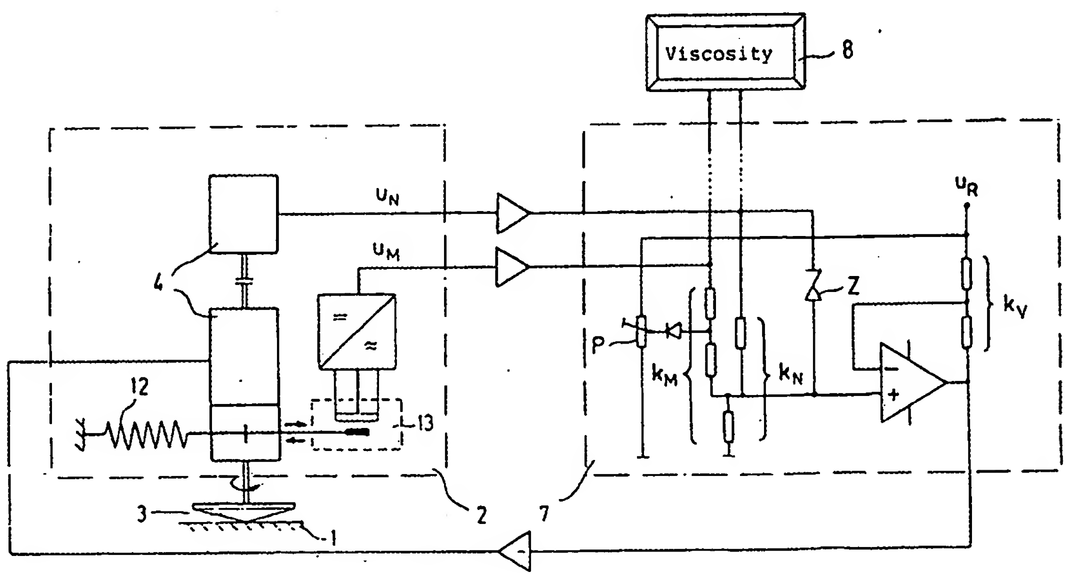

FIG. 2

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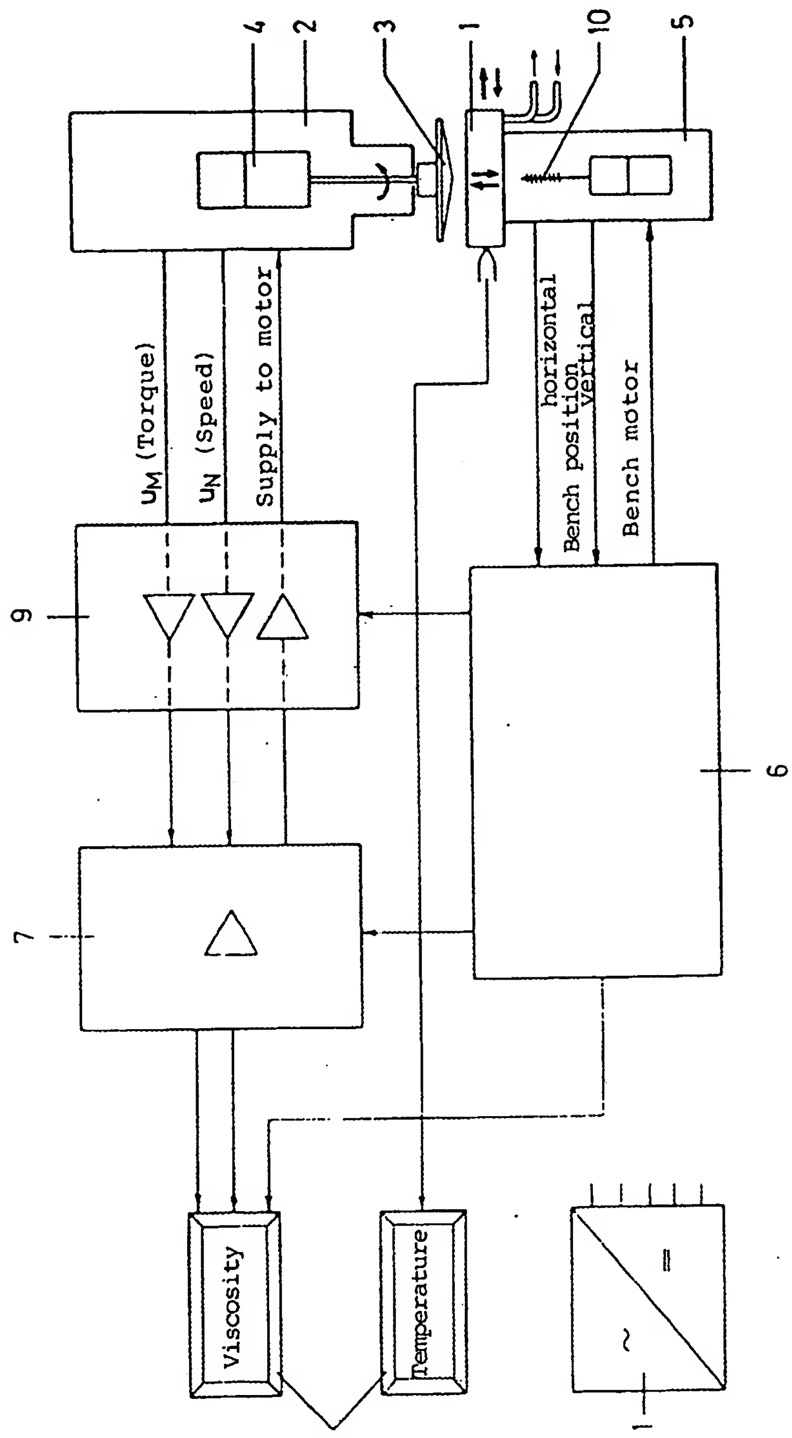
4
2
3
1
10
5
U_M (Torque)
U_N (Speed)
Supply to motor
horizontal
Bench position
vertical
Bench motor
9
6
7
Viscosity
Temperature
8
11

FIG. 1

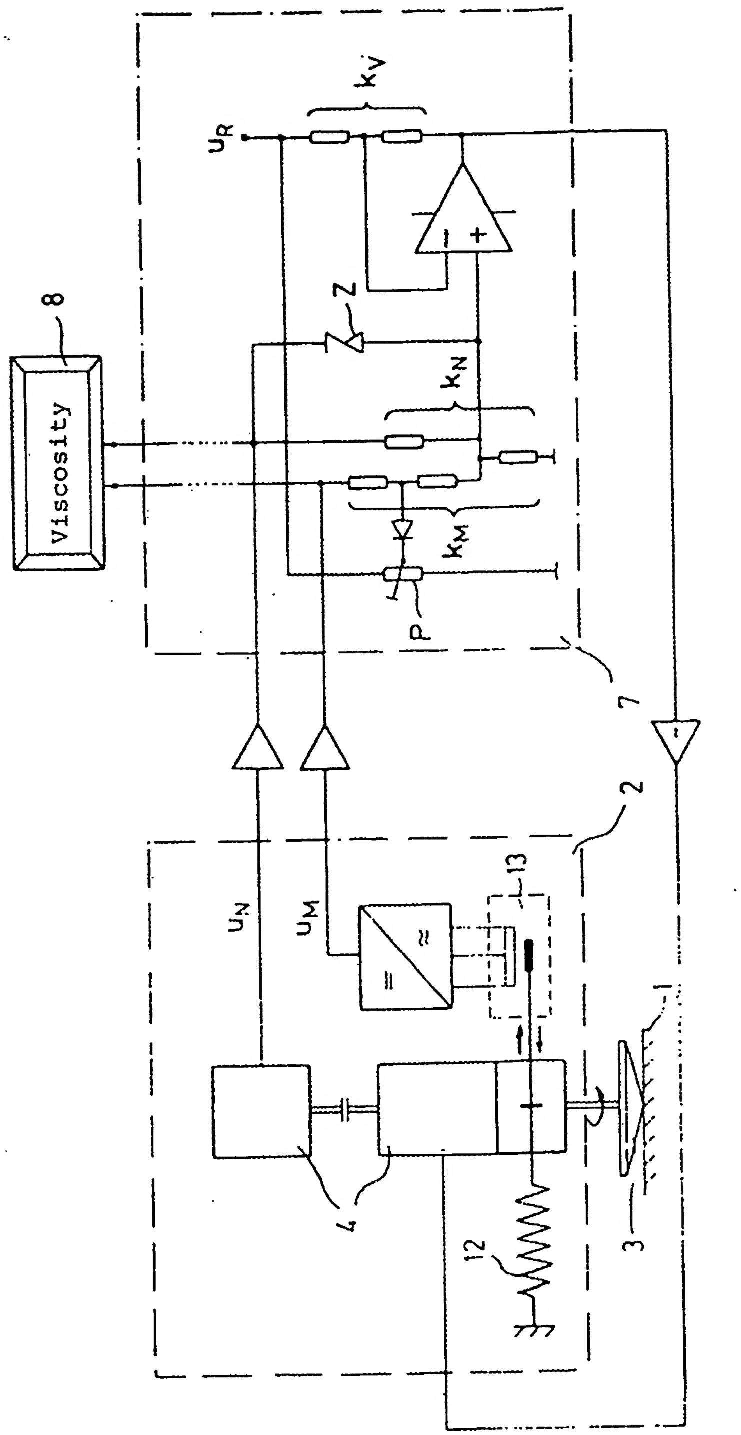

FIG. 2

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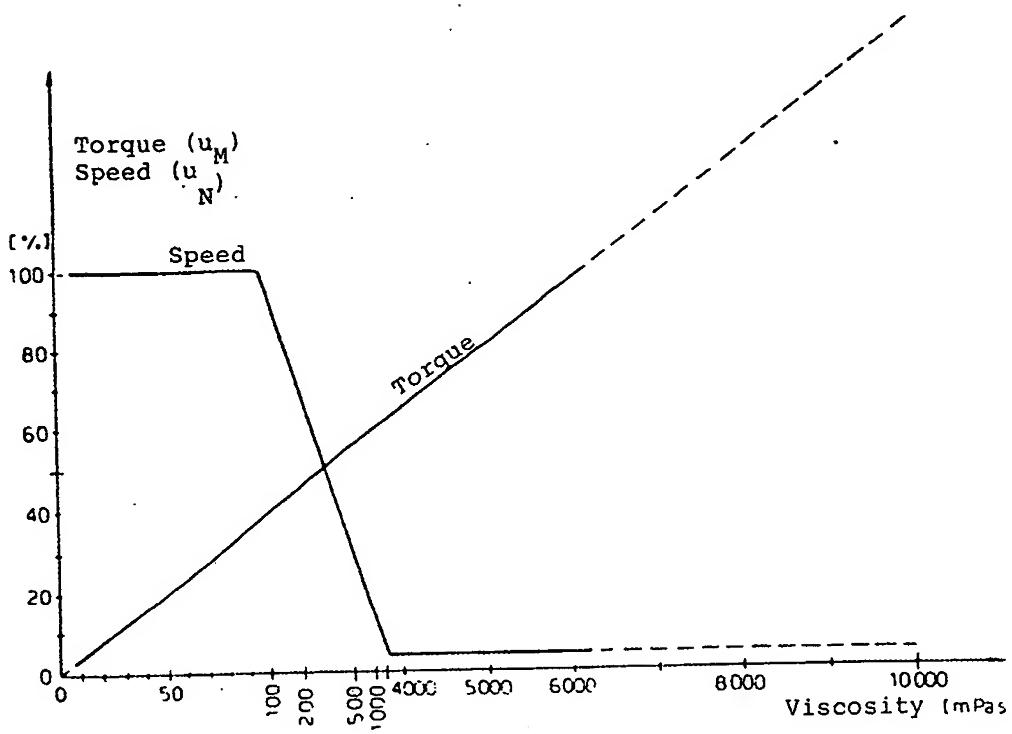


FIG. 3

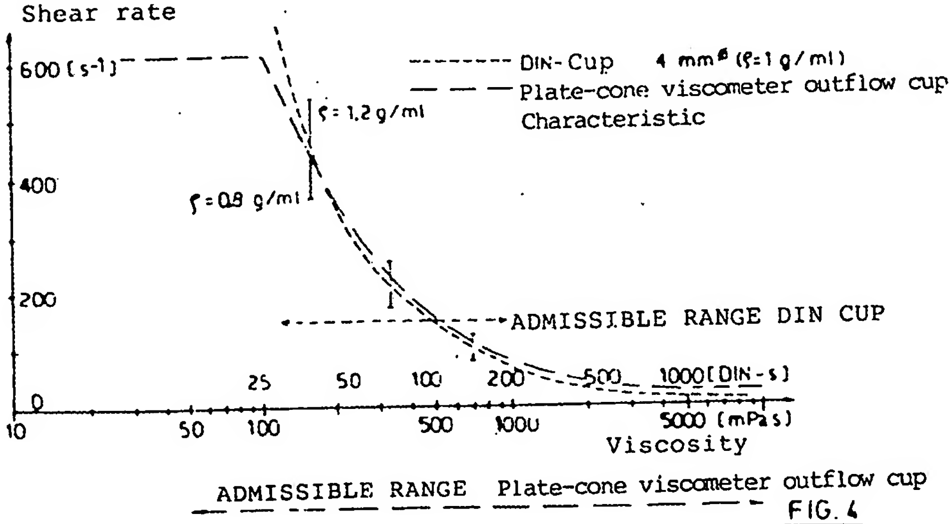
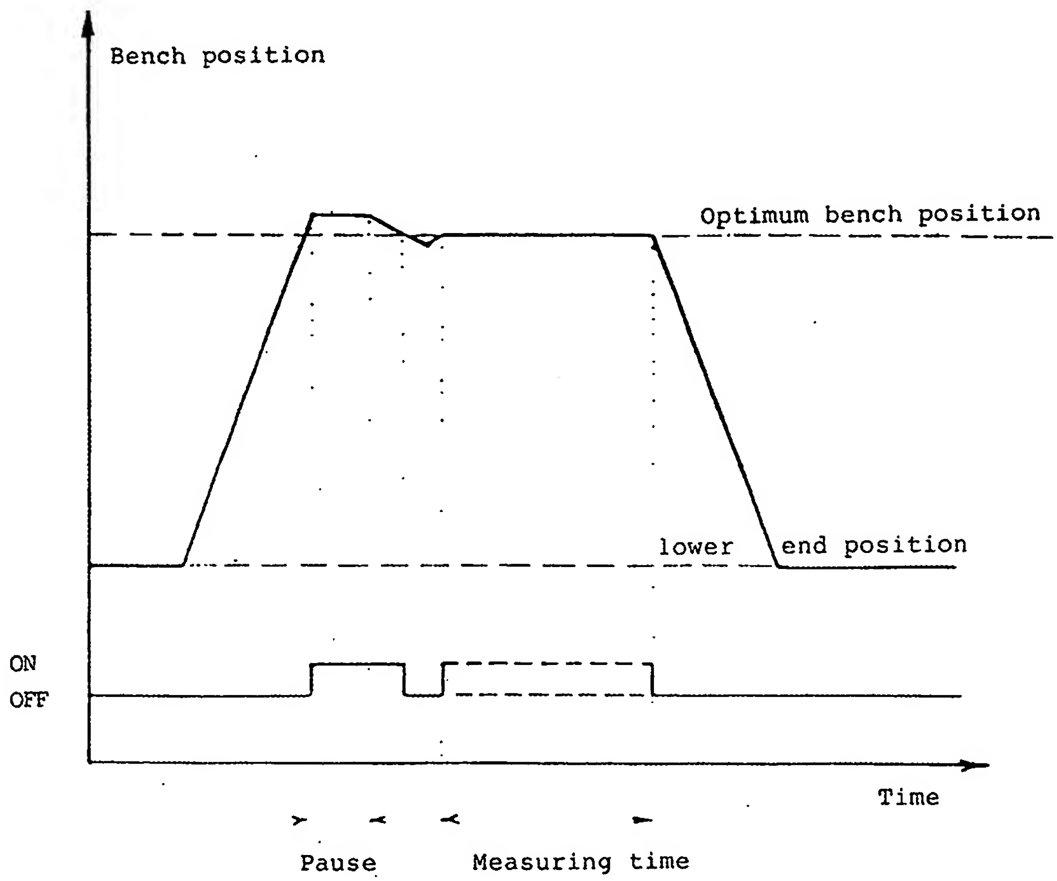


FIG. 4



Bench position
Optimum bench position
lower end position
ON
OFF
Time
Pause
Measuring time

FIG. 5

SPECIFICATION

Plate-cone viscometer with viscosity-dependent regulation of shear rate

In conventional plate-cone viscometers, the dynamic viscosity is determined whilst taking into account various instrument constants or calibrating factors. This can be done with various cone angles or diameters and speeds. Thus, when the flow characteristics of the samples are not (strictly) Newtonian, different measurements may be obtained on the same sample. Instruments of this kind are suitable for difficult measuring procedures but not for continuous routine measurements in a production run in which samples of very different viscosity have to be measured.

Plate-cone viscometers typically have a measuring dynamic range of 1:3, with adjustment of the speed and without changing the cone geometry, if the error in measurement is to remain below ±1%.

However, in production monitoring such as is carried out in the manufacture of alkyd or polyester resins, viscosity ranges of at least 1:10 to 1:20 must be measured rapidly and reproducibly. Multiple measurements made necessary by manual switching of the range ("trial and error") would be liable to error: the evaporation of volatile solvent components makes it essential to have short measuring times and the smallest possible surface areas exposed to the air. In conventional viscometers there is also the risk of errors if a number of conversion factors have to be included in calculations.

Therefore, the outflow cups according to DIN 53211, which are simple to handle, are in widespread use; they do have a useful viscosity measuring range of 1:10, but owing to the large sample quantity (100 ml) they require long times to be brought to the correct temperature (about 20 minutes) and suffer further inaccuracies of measurement caused, *inter alia*, by air bubbles stirred in.

There is therefore a tendency to switch production control to other methods. For reasons of comparability the same viscosity-dependent shear rates have to be maintained when such a changeover is made.

It has now been found that by suitable adaptation of standard commercial plate-cone viscometers, sequential measurement of dynamic viscosity can be carried out in a production run without the usual complicated operation and evaluation.

The invention th refore relates to a plate-cone viscometer with viscosity-dependent regulation f the shear rate for simple sequential viscosity measurement, consisting of a measuring head which contains a replaceable measuring cone and a drive motor with tachogenerator and a bench capable of being brought to a desired temp rature and adjustable in position the bench having a plate connected to it, characterised in that the plate together with the bench are brought into the optimum position relative to the measuring cone by means of a motor-driven micrometer spindle and a process control apparatus for each measurement, the measuring position being signalled by means of electrical contacts and, during measurement, the drive motor for the measuring cone being controlled by the output signal from an analog computer inserted in a regulating circuit, this analog computer linking the electrical signals for the metal torque (u_M) and speed (u_N) continuously and in weighted manner according to the equation

$$u_N \cdot k_N + u_M \cdot k_M - u_R \cdot k_V = 0,$$

wherein u_M is a voltage proportional to the torque, u_N is a voltage proportional to the speed and u_R is a reference voltage and k_M, k_N and k_V are coefficients which determine the weighting.

Preferably the voltages u_N, u_M and u_R are between -15V and $+15$V and the coefficients k_N, k_M and k_V are in the range from 0.01 to 20.

Compared with the conventional method of determining viscosity by measuring the outflow time using an outflow cup (e.g. according to DIN 53211), this significantly reduces the time for reaching the correct temperature and making the measurement gives increased accuracy, reduces the quantity of sample required to 1/100 and expands the measuring range.

An embodiment of the invention will now be described by way of example.

Figures 1 to 5 show the following:

Fig. 1 is a schematic view of the basic construction of the apparatus claimed. The reference numerals denote the following:

(1) Measuring plate
(2) Measuring head
(3) Measuring cone
(4) Drive motor with tachogenerator
(5) Test bed with plate (1), capable of being brought to a desired temperature and swung out laterally
(6) Position and process control
(7) Analog computer for continuous regulation of the shear rate
(8) Indicator instruments for the bench temperature (°C) and the measurement obtained for the dynamic viscosity
(9) Supply device for the drive assembly
(10) Motor-driven micrometer spindle for the plate (1)
(11) Mains supply

Fig. 2 schematically shows the analog computer which links the signals from the measuring head for the torque and speed to one another continuously and in weighted manner and controls the motor so as to obtain a transmission function as shown in Fig. 3. The

linking is in accordance with the equation

$$u_N \cdot k_N + u_M \cdot k_M - u_R \cdot k_V = 0,$$

wherein u_N is the voltage proportional to the speed of the cone and u_M is the voltage proportional to the torque and u_R is a reference voltage, k_M is the coefficient for the torque, which determines the weighting, and k_N is the coefficient for the speed and k_V is the coefficient for the amplification.

By a suitable choice of dimensions for the components and by a suitable choice of coefficients for this analog computer it is possible, for example, to simulate the dependency of the average shear rate on the viscosity as in the measurement of the outflow time according to DIN 53211 (see Fig. 4), thereby obtaining directly comparable values.

In a practical embodiment the voltages u_N, u_M and u_R are between −15V and +15V and the coefficients k_N, k_M and k_V are in the range from 0.01 to 20 and preferably the coefficients k_M, k_N and k_V have the following values: k_M = 0.5 to 1.2, k_N = 0.1 to 0.3, k_V = 0.7 to 1.5.

The reference numerals and symbols used in Fig. 2 have the following meanings:

4 Drive motor with gears and tachogenerator
12 Measuring spring
13 Non-contact distance measurement
k_M Coefficient for the torque
k_N Coefficient for the speed
k_V Coefficient for the amplification
P Potentiometer for setting the minimum speed
Z Zener diode for limiting the maximum speed
u_M Voltage proportional to the torque
u_N Voltage proportional to the speed
u_R Reference voltage

Fig. 3 shows, by way of example, a transfer function such as is obtained by using the analog computer (7) for the continuous regulation of shear rates, the useful measuring range extending from about 50 to 10,000 mPas.

Fig. 4 shows, by way of a comparison, the good comparability of the apparatus to the shear rates which occur with the DIN cup.

Fig. 5 shows the sequence of events in the automatic positioning of the bench as a function of the signal which indicates contact between the plate and cone ("ON", "OFF").

Since plate-cone viscometers theoretically function correctly only when the cone is just touching the plate, the greatest care must be taken in adjusting the b nch (= plate). When the tip of the cone presses onto the bench, an additional frictional moment is produced which falsifies the measurement and makes the cone unusable through wear. If there is a gap between the tip of the cone and the bench, the viscometer indicates too little, and depending on the geometry of the cone gaps of a few microns may lead to errors of measurement of several percent.

In the proposed apparatus, therefore, a motor-driven micrometer spindle (10) is used for movement of the bench and this micrometer spindle, cooperating with the process control means (6), seeks the optimum measuring point afresh for each sample.

The signal indicating whether the plate is touching the cone is produced by alternating-current voltage having a frequency in the range 10 Hz to 100 KHz and preferably in the audio frequency range in order to prevent polarisation or decomposition effects in the sample; the time sequence of this signal is shown in Fig. 5.

The test bench (5) has thermostatically controlled fluid flowing through it for bringing it to a desired temperature and is set precisely at right angles to the cone axis by means of adjustment screws. Moreover, the entire bench can be pivoted sideways out of the axis of the sample to facilitate the cleaning of the plate and cone.

The position and process control means (6) control and monitor all the processes in the positioning of the test bench and in the measurement and calibration of the apparatus. As shown in Fig. 5, the flow of highly viscous samples as a function of time as the plate approaches the cone is taken into account when positioning the test bench.

The control means used consist of freely programmable control means based on a microprocessor fitted with slow, interference-free inputs and relay outputs.

For monitoring production in the manufacture of alkyd, polyester or copolymers, used for example as lacquer binders, an apparatus having the following technical data may be used, for example:

Cone used: Diameter 50 mm
Angle (relative to the plane of the bench): 1 degree
Maximum shear rate: about 600 s^{-1}
Minimum shear rate: about 25 s^{-1}
Viscosity measuring range (with the cone specified above):
for maximum accuracy: 100—2,000 mPa.s
for reduced accuracy: 50—10,000 mPa.s
(For comparison, an outflow time of 25 to 200 seconds using the DIN outflow cup 4/20°C (DIN 53211) corresponds to values of from 100 to 1,000 mPa.s.)
Resolution for visc sity: ± 1 mPa.s
Reproducibility: ± 2%
Admissible zero-p int error: ± 0.15% of the maximum torque
Resolution for temperature: 0.1°C
Electrical contact between plate and cone: measuring voltage: max. 1.5 V_{pp} (about 2 kHz)
Typical measuring time: 50 seconds (including reaching desired temperature, no cleaning)

CLAIMS

1. Plate-cone viscometer with viscosity-dependent regulation of the shear rate for simple sequential viscosity measurement, consisting of a measuring head which contains a replaceable measuring cone and a drive motor with tachogenerator and a bench capable of being brought to a desired temperature and adjustable in position the bench having a plate connected to it, characterised in that the plate together with the bench are brought into the optimum position relative to the measuring cone by means of a motor-driven micrometer spindle and a process control apparatus for each measurement, the measuring position being signalled by means of electrical contacts and, during measurement, the drive motor for the measuring cone being controlled by the output signal from an analog computer inserted in a regulating circuit, this analog computer linking the electrical signals for the metal torque (u_M) and speed (u_N) continuously and in weighted manner according to the equation

$$u_N \cdot k_N + u_M \cdot k_M - u_R \cdot k_V = 0,$$

wherein u_M is a voltage proportional to the torque, u_N is a voltage proportional to the speed and u_R is a reference voltage and k_M, k_N and k_V are coefficients which determine the weighting.

2. A plate-cone viscometer as claimed in claim wherein the bench is laterally pivotable.

3. A plate-cone viscometer as claimed in claim or 2 wherein the voltages u_N, u_M and u_R are between $-15V$ and $+15V$.

4. A plate-cone viscometer as claimed in claim 1, 2 or 3 wherein the coefficients k_N, k_M and k_V are in the range from 0.01 to 20.

5. A plate-cone viscometer as claimed in any preceding claim wherein the coefficients k_M, k_N and k_V have the following values: k_M = 0.5 to 1.2, k_N = 0.1 to 0.3, k_V = 0.7 to 1.5.

6. A plate-cone viscometer as claimed in any preceding claim, characterised in that the signal indicating contact between the plate and the measuring cone is produced by alternating-current voltage having a frequency in the range from 10 Hz to 100 kHz.

7. A plate-cone viscometer as claimed in claim 6 wherein said frequency is in the audio frequency range.

8. A plate-cone viscometer substantially as hereinbefore described with reference to the accompanying drawings.

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